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SEAL MATERIAL DEVELOPMENT TEST PROGRAM

FINAL REPORT

PREPARED FOR
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
MANNED SPACECRAFT CENTER
HOUSTON, TEXAS 77058

CONTRACT NO. NAS 9-11866

TRW
SYSTEMS GROUP

OFFICE OF PRIME RESPONSIBILITY

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NASA MANNED SPACECRAFT CENTER

HOUSTON, TEXAS

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FOREWORD

This report presents the results of a program to characterize the properties of an experimental fluorinated elastomer AF-E-124D as a potential seal material. This program is an extension of a previous program, Seal Material Development, Contract NAS 9-10481, in which the material AF-E-124D was identified as a promising seal material for liquid oxygen/liquid hydrogen propulsion systems. The program was initiated in June 1971, and conducted by the Advanced Technology Division of TRW Systems, Redondo Beach, California, for the NASA Manned Spacecraft Center, Houston, Texas under Contract NAS 9-11866. The program was conducted under the technical direction of Mr. M. C. Buchanan of the NASA Manned Spacecraft Center. Mr. F. E. Compitello was the NASA Headquarters Project Manager. The TRW Systems Program Manager was Mr. R. G. Gilroy. Program activities were conducted at TRW Systems, Redondo Beach and TRW Capistrano Test Site, Capistrano, California.

AF-E-124D material was used with the approval and assistance of the Air Force Materials Laboratory, Wright-Patterson Air Force Base, Dayton, Ohio.

Substantial support and assistance was provided by the Applied Chemistry Department, Dr. E. A. Burns, Manager, which molded the AF-E-124D material. Acknowledgement is also made of other major contributors to this program: Mr. J. W. Martin for materials and technical assistance, Mr. J. R. Denson for testing, and Mr. H. W. Wright and Mr. G. W. Howell for technical assistance, report preparation and data evaluation.

ABSTRACT

This program, Contract NAS 9-11866 Seal Material Development Test Program was conducted to characterize an experimental fluoroelastomer material, AF-E-124D. This material was identified as a potentially superior seal material in a previous program under Contract NAS 9-14081 (reported in TRW Report 14771-6001-RO-00). The previous program was conducted to identify and characterize materials which would advance the state-of-the-art for oxygen-hydrogen propulsion system seals. AF-E-124D was partially characterized in that program along with eighteen other polymeric seal materials, over a temperature range of +200°F (600°R) to -423°F (37°R).

This program represents a continuation of the previous program, specifically oriented toward more complete characterization of AF-E-124D. Tests conducted include liquid nitrogen load compression tests, flexure tests and valve seal tests, ambient and elevated temperature compression set tests, and cleaning and flushing fluid exposure tests. The results of these tests indicate that AF-E-124D is a good choice for a cryogenic seal, since it exhibits good low temperature sealing characteristics and resistance to permanent set.

The status of this material as an experimental fluorelastomer is stressed and recommended activity includes definition and control of critical processing to ensure consistent material properties. Design, fabrication and test of this and other materials is recommended in valve and static seal applications.

CONTENTS

APPENDIX

ILLUSTRATIONS

TABLES

1. SUMMARY

The Seal Material Development Test Program was conducted to characterize the attributes of the experimental perfluorinated elastomer AF-F-124D. A previous program, "Seal Material Development, Phase I," was conducted to identify materials which would significantly advance the state of the art in seals for oxygen-hydrogen propulsion systems. This requires materials compatible with hydrogen and oxygen at temperatures from $-423^{o}F$ ($37^{o}R$) to $+200^{o}F$ ($660^{o}R$). The mechanical properties must be such that the parameters important to seal materials are retained over this temperature range. Consequently, the superior ambient and elevated temperature sealing capability of elastomers strongly indicated that this program be oriented toward identifying elastomers with acceptable low temperature sealing and mechanical properties.

The previous program identified a single material which exhibited the desired sealing properties and, in general, was superior to the control material Teflon with both liquid hydrogen and liquid oxygen. The current program was formulated to further characterize the properties of AF-E-124D.

The tests performed in this program were: LN_2 load compression tests, ambient and elevated temperature compression set tests, LN_2 flexure tests and LN_2 valve seal tests. A series of solvent exposure tests was conducted to establish the compatibility of the material with commonly used cleaning and flushing fluids. These tests indicate that AF-E-124D is a good choice

for a cryogenic seal, exhibiting good low temperature sealing capability and resistance to permanent deformation. Load compression testing with bulk material in LN_2 indicated a permanent set after a cryogenic cycle, approximately equal to Teflon, but with a much higher LN_2 load retention capability when cooled to LN_2 temperature. Required sealing loads at LN_2 temperature are approximately 32% less than Teflon with the same ratio of load required between ambient and cryogenic temperature (approximately a factor of 3). Solvent exposure tests indicate that AF-E-124D is compatible with distilled water and isopropyl alcohol for periods up to 14 days (which was the maximum test duration) and much less compatible with Freon TF and trichlorethylene.

The AF-E-124D material evaluated in this program is a base polymer with no fillers or other additives. Continuing development of the material by TRW on other programs includes the compounding with other additives to establish which properties can be further improved. The material tested is a "base" polymer from which a series of compounds may be developed.

Although the objective of this program was to characterize AF-E-124D specifically to use with hydrogen and oxygen over a temperature range from cryogenic to elevated temperatures, the applicability of this material as a seal for even a broader range of propellant applications is of significant interest. In that light data are presented on the compatibility of AF-E-124D with storable propellants. Based on favorable compatibility data with ClF_3, N_2H_4 and N_2O_4, the perfluoroelasomer shows the potential of being a universal seal material.

The processing of AF-E-124D is at present very critical and slight processing variations will result in different material properties and varying test results. Recommendations for future effort include definition and control of the critical processing parameters, and the fabrication of seals for valve testing. Also recommended is continued investigation of valve and static seals of Viton, HYSTL and Teflon.

2. INTRODUCTION

The objective of this program was to supplement the information obtained during the previous program, (Seal Material Development Program, Contract NAS 9-10481)[1] identifying advanced cryogenic seal materials in general, and characterizing AF-E-124D in particular. During the previous program, a number of polymeric seal materials were evaluated for their applicability as a cryogenic seal material. Late in the previous program an experimental perfluorinated elastomer identified as AF-E-124D was available and preliminary screening tests were conducted. On the basis of these limited tests, this new material appeared very promising. The current program was designed to further characterize the AF-E-124D material by additional testing.

This report relates the work accomplished under the present contract to the previous results. The program conclusions are identified with recommendations for further work in cryogenic seal development such as seal design evaluation and application to components such as valves.

3. PROGRAM DESCRIPTION

PROGRAM REQUIREMENTS AND CONTENT

This program was planned specifically to provide the degree of characterization for seal material AF-E-124D that was provided for other materials in the previous program under Contract NAS 9-10481. During that program the basic approach was to primarily investigate elastomeric materials because of the generally superior sealing characteristics of elastomers at ambient and elevated temperatures, where the majority of seal degradation occurs. From that program, AF-E-124D appeared to have the characteristics for a seal material for both liquid oxygen and liquid hydrogen service.

The tasks conducted in this program were:

Task I Liquid Nitrogen Testing

- Load Compression Tests
- Flexure Tests
- Valve Seal Tests

Task II Solvent Exposure Tests

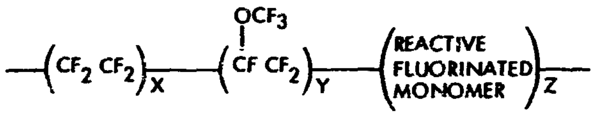
Task III Evaluation and Correlation of Data
Final Report

All testing was conducted with AF-E-124D, using Teflon TFE as the control.

4. CHARACTERISTICS OF AF-E-124D

4.1 COMPOSITION

The elastomer evaluated during this program has been identified as AF-E-124D, a proprietary development material supplied by the Air Force Materials Laboratory, Elastomers and Coatings Branch to TRW Systems for compounding and process studies under Contract F33615-71-C-1233. While not verified, the material has certain characteristics suggesting that it is similar to ECD-006, which has been described in the open literature.[2] Using this literature as a source, it can be speculated that AF-E-124D is a terpolymer of tetrafluoroethylene, methyl vinyl ether and a fluorinated monomer used as a crosslinking site shown schematically in Figure 4-1. The resulting polymer is fully fluorinated (or perfluorinated).



WHERE X > Y > Z, AND DISTRIBUTION IS RANDOM

Figure 4-1. Speculated Composition of AF-E-124D

In contrast to other fluorinated elastomers which are not fully fluorinated, the perfluorinated elastomers are closely related to the Teflon polytetrafluoroethylene (TFE) family of plastics. The combination of the chemical inertness and thermal stability of Teflon, combined with the resilience and compliance of an elastomer makes the perfluorinated elastomers attractive as seal materials. Flexibility if built into the polymer by the pendant methyl ether groups, without significantly decreasing the chemical resistance of the molecule. Well known is the creep or "cold flow" of TFE relative to thermosetting elastomers and this apparently has been decreased in AF-E-124D by introduction of a relatively reactive monomer into the polymer to provide a chemically stable site for curing. Linear polymers such as TFE, other thermoplastics and uncured elastomers are susceptible to stress induced creep as the polymer molecules can slip over one another. Crosslinking reactions develop a three dimensional matrix of chemically bonded molecules which reduces slippage considerably. Nature of the cure site monomer is proprietary, but it is believed labile to nucleophilic attach by a chemical cure agent supplied with the base stock. Concentration of the cure site monomer also is not known, but typical crosslink termonomers of elastomeric polymers are in the order of 2 to 5 mole-% and it is speculated that the cure site monomer of AF-E-124D is in this range of concentration.

Nucleophilic reaction at the crosslinking site during cure is a relatively high kinetic energy process, favored thermodynamically. This conclusion is drawn from the extensive time required to develop the full cure of the polymer at high temperatures.(3) It can be further speculated that the nucleophile is in high concentration due to the high shrinkage during oven post cure which is generally recognized as a period of purging the spent cure agent.

Perfluorinated polymers have no carbon-hydrogen bonds. Instead, the more stable carbon-fluorine bonds (by about 8 to 10 kcal/mole) are present which result in the overall stabilization of the polymer both to reducing agents (in this instance, fuels) and oxidizing agents (such as N_2O_4 or LO_2). AF-E-124D chemically is like TFE, but with more elastomeric properties imparted by a more flexible polymer chain and with decreased creep due to crosslinking.

4.2 PROCESSING

AF-E-124D is a rubber by definition, but processes more like a thermoplastic. At best, it is difficult to process. The material must be processed on a heated (160-180°F) two-roll rubber mill and is difficult to band until it has become heated thoroughly. Once banded to form a semi-liquid rather than a crumbly solid, the material must be sheeted off to approximately its eventual thickness, prior to molding. It must be transferred from the heated mill to the preheated mold (about 300° to 350°F) rapidly before it cools. When pressed in the preheated mold at several thousand pounds per square inch, it will flow in the cavity, but not readily. Such an observation is difficult to quantitate, but the surface area of a preform can be increased only 10 to 20% during the molding operation, which is most typically 30 minutes at 350°F.

About 75% of the tensile strength is developed during the press cure and this is increased further by curing in an air oven following a schedule which is complex compared with conventional elastomer cure cycles.

A general rule is that the material should be cured to at least 50°F above its projected use temperature. Thus, for 350°F service, the cure could be stopped after the 400°F step. Full cure of AF-E-124D is reached after 24 hours at 550°F.

After full cure, nearly 10% shrinkage is found for AF-E-124D. This must be considered when fabricating complex or molded-in-place seals. This problem was experienced to some degree in molding the load compression test samples for this program. Trimmed-to-fit seals appear to be more feasible at this time.

One property which impacts strongly the potential usefulness of AF-E-124D as a dynamic seal is its surface characteristics after curing. The gross surface is often rough due to molded-in-stresses and poor flow. However, the micro surface is very smooth and the material has very strong self-adhesion and much greater than usual adhesion to other smooth surfaces. Processing studies are currently in progress at TRW Systems to find suitable additives for the polymer which will improve the mold flow, decrease the cure shrinkage, but not impair the chemical stability of the polymer.

4.3 MECHANICAL PROPERTIES

AF-E-124D has been reported to be a non-reinforced polymer, that is, without added particulate fillers intended to increase the polymer's strength properties. If true, this is a polymer which reaches high strength in a gum compound like natural rubber (CIS 1,4-polyisoprene) or chloroprene, both of which are strengthened by strain-induced crystallization. However, this property of AF-E-124D is not altogether illogical when it is realized that a majority of the polymer is TFE, which also reaches high strength without crosslinking. At relatively high strain rates, the crosslinked material has excellent rubber-like properties. At lower strain rates over longer periods, the material shows a plastic-like behavior. A strong sensitivity to tension shear rate[5] and to compression creep is displayed by the crosslinked polymer. This behavior of AF-E-124D is another property like that of TFE. Properties of AF-E-124D are shown in Table 4-1. These data also indicate a type of variability in properties which can result from minor processing differences. The base compound and cure were identical in each case, the differences in properties apparently being caused by processing differences.

Table 4-1. AF-E-124D Mechanical Properties

	Sample 1**	Sample 2**	Sample 3**
Hardness, Shore	80	69	78
Tensile Strength* at Break, psi	2520	2335	2600
Modulus* at 100%, psi	890	910	835
Elongation* at Break, %	160	155	150
Tensile Set, %	8	3	2

*Tested at 72°F with a cross head speed of 20 inch/min. (ASTM Standard)

**Average of triplicate samples

Addition of reinforcing fillers such as silica or carbon black is used to reduce the compression set of gum rubber. With AF-E-124D, there appears to be an undesirable chemical reaction between silica and the polymer at high temperature. Carbon black supplies the necessary mechanical reinforcement at the possible cost of chemical inertness. Fluoride salts appear promising as reinforcement agents for the polymer but these have not been evaluated thoroughly and no firm conclusions are practical at this time

4.4 THERMAL PROPERTIES

The low temperature properties of a polymer are largely a function of its glass transition temperature (T_g). This temperature is the point at which a polymer undergoes a transition to become hard and rigid. This point is above -200°F for all known polymers. Below this temperature the general physical properties of a material do not change as rapidly with decreasing temperature. Some slight modification in this useful temperature can be brought about by decreasing the crosslink density, the use of plasticizers and other compounding variables, but the T_g of a random copolymer is determined largely by the monomer species used and the ratio of these monomers. Also, these properties are affected by crystallization. The T_g of AF-E-124D is about +3°C (37.5°F) which for a conventional elastomer compound would also represent the low temperature useful limit of the material. AF-E-124, however, is not a conventional elastomer.

AF-E-124D has useful engineering properties without using particulate inorganic fillers. It was observed during the Phase I testing that generally "neat" polymers, that is, those without particulate inorganic fillers, showed good sealing ability at low temperature, and that this may be a result of their capability to retain some degree of flexibility below their T_g. This is true of Mylar, TFE, FEP, polyethylene, HYSTL (1,2-polybutadiene resin) and AF-E-124D. It is speculated that other neat polymers also would remain flexible in compression below their T_g. Most other polymers require particulate inorganic fillers to generate acceptable mechanical and engineering properties required of a seal resulting in increased brittleness at low temperature. The polymers listed above all have in common that they are strong without particulate fillers and seal at temperatures well below their T_g. On this basis, a major finding of this program, i.e., that AF-E-124D seals at temperatures well below its T_g, is consistent with expectations. A possible compromise that will result is that in adding reinforcing fillers to decrease shrinkage during cure, and to decrease compression set, some degradation in low temperature sealing may result. As indicated earlier, processing studies are currently in progress at TRW to determine suitable additives. The next step would be to establish the effect of these additives on low temperature sealing capability.

At high temperatures, AF-E-124D is quite resistant to oxidation. This property is a direct consequence of the carbon-carbon single bonds comprising the backbone of the polymer and of the stabilizing effect of carbon-fluorine side bonds. Polymer degradation primarily is a function of backbone stability. Side chain ether groups contain the relatively stable carbon-oxygen and carbon-fluorine bonds, and thus the entire polymer is resistant to oxidation.

It should be pointed out that most elastomers have a coefficient of thermal expansion at least one order of magnitude greater than typical metals. Most conventional rubber compounds are 30-60% by weight base polymer and the remainder is fillers with lower thermal expansion coefficients. AF-E-124D apparently is a neat base polymer and therefore will probably have a relatively high coefficient.

5. TEST DESCRIPTION AND RESULTS

5.1 LIQUID NITROGEN TESTS

This series of tests was conducted similar to those performed during the Phase I program. The tests run were:

- Load Compression
- Flexure Tests
- Valve Seal Tests

5.1.1 Load Compression Tests

These tests were conducted to identify the load retention capability of AF-E-124D at low temperature and subsequent degree of recovery after return to ambient temperature. This property is of primary importance for static seal applications.

The test sequence used is shown schematically in Figure 5-1.

A schematic of the test set-up is presented in Figure 5-2. The test procedure is as follows. Each specimen height is accurately measured prior to test. After initially bringing the ram into contact with the sample, the reference mark is sighted, zeroed, and the load cell zero point recorded. Load is applied by torquing the ram. When the planned deflection of approximately 25 percent is obtained, the load required to achieve this compression is recorded and the load is then allowed to stabilize at room temperature. The assembly is then cooled to -320°F by filling the Dewar flask with LN_2 to a level above that of the test specimen. After temperature stabilization, the residual load of the test sample is recorded from the load cell. The assembly is then allowed to warm-up to room temperature, the residual load recorded, then removed by untorquing the ram. The final deflection measurement is recorded at the zero load point. From this test sequence, the following data are obtained:

(1) Residual load at LN_2 temperature

(2) Preload loss at room temperature after a cycle from room temperature to -320°F to room temperature

(3) Permanent set as a result of one temperature cycle

TEST SEQUENCE

1. LOAD APPLICATION
2. STABILIZATION
3. COOL DOWN
4. WARM-UP
5. LOAD REMOVAL

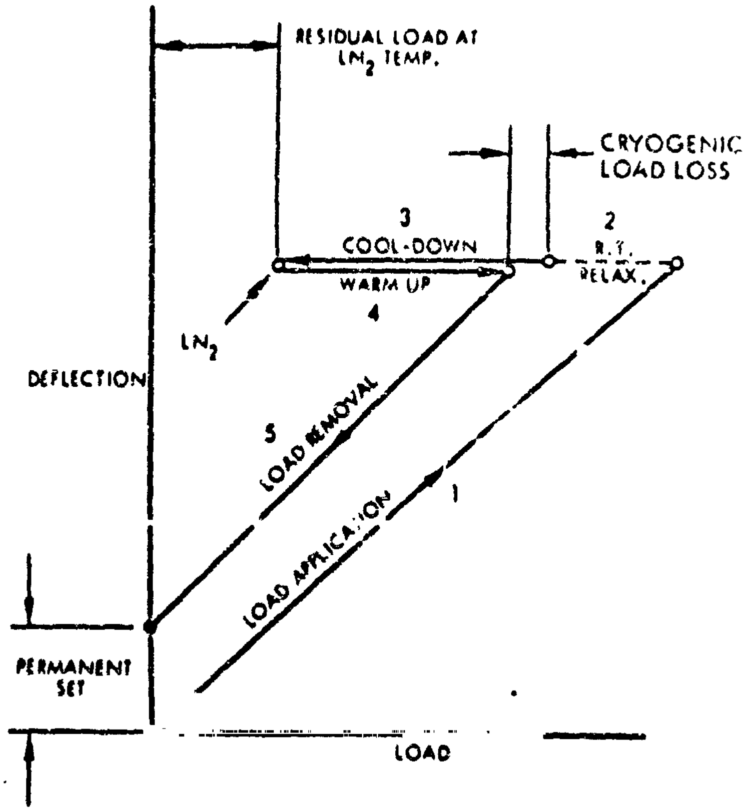


Figure 5-1. Idealized Load-Deflection Cryogenic Cycle

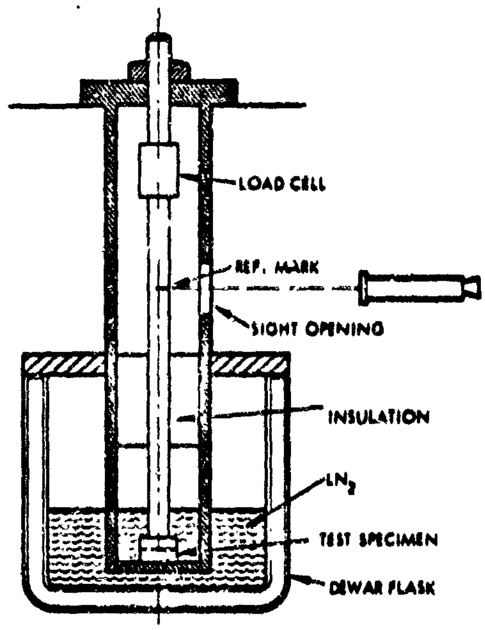


Figure 5-2. Test Fixture Schematic

The test data for AF-E-124D are plotted in Figure 5-3. This curve shows the typical elastomer load compression curve and relaxation of load after some period at room temperature. Other compression data were generated to better characterize room temperature and elevated temperature characteristics. They are presented later.

The permanent compression set of AF-E-124D resulting from a single LN_2 cycle is approximately 5 percent at an initial preload of 22 percent. Load retention at LN_2 temperature is approximately 60 percent which compares with approximately 33 percent for Teflon TFE.

Table 5-1 shows data presented in the Phase I report with AF-E-124D information added. In general, the properties are equal or superior to those of Teflon and superior to the others.

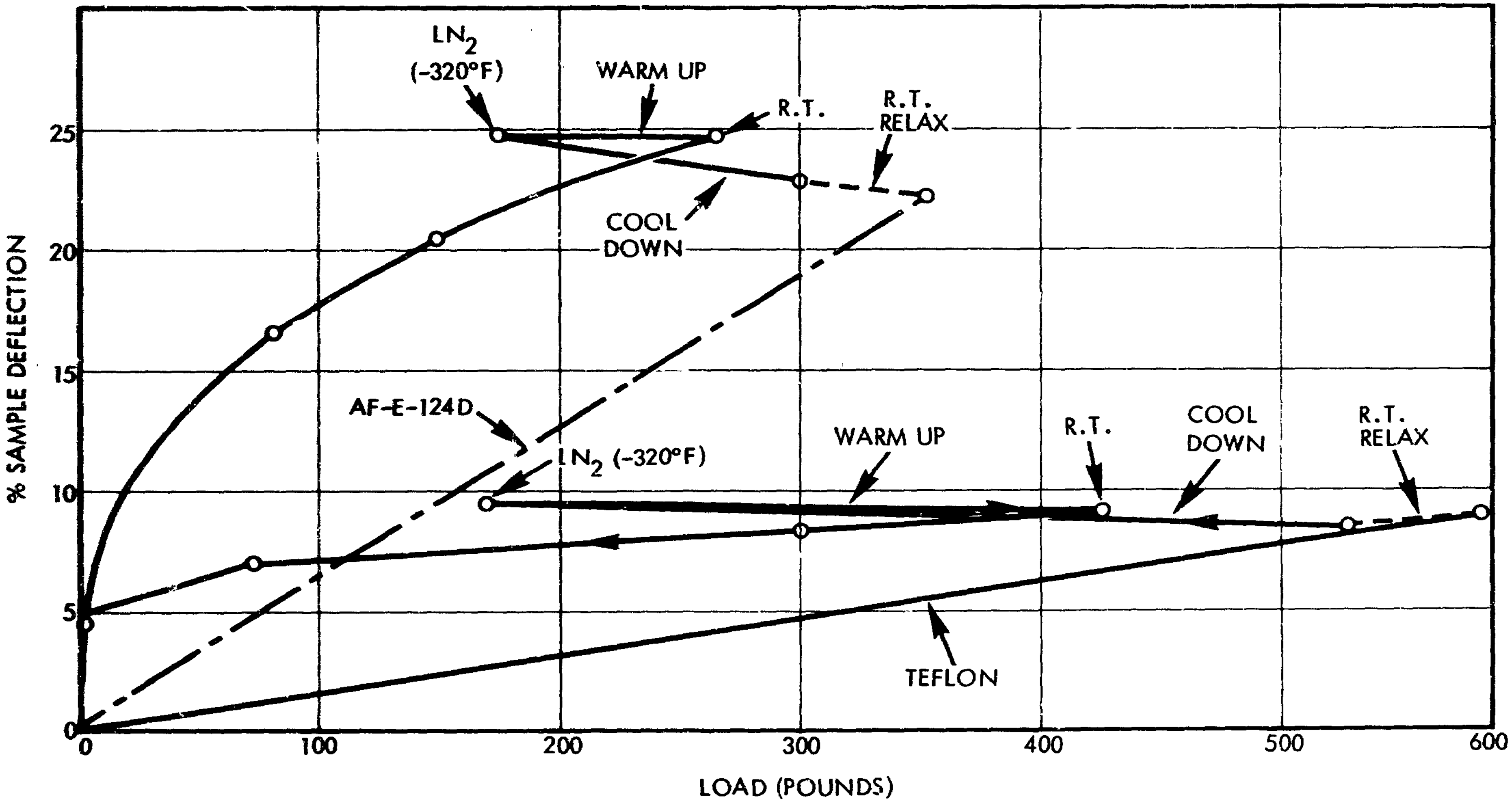


Figure 5-3. Load Deflection LN_2 Temperature Cycle AF-E-124D and Teflon TFE

Table 5-1. Compression Load Test Data*

Material	Compression (%)	Final Permanent Set (%)	Loads				Load Retention		
			Initial Room Temperature (lbs)	Relax Room Temperature (lbs)	LN_2 Temperature (lbs)	Recovery Room Temperature (lbs)	Room Temperature Relaxed/Initial (%)	LN_2 Temperature (% of Relax Load)	Room Temperature Recovery (% of Relax Load)
Viton-A (255-2)	27	2.6	284	175	67	175	62	38	100
EPT-NYSTL (AF-E-71-2)	22	1.4	592	440	142	421	74	33	96
EPT-HYDRIN (263-3)	25	3.8	577	462	128	440	80	27	96
Buna-N (215-2)	12	1.7	104	46	6	43	44	13	94
Polybutadiene (60-7)	26	1.2	269	208	6	180	78	3	86
Phenylsilicone (310-1)	24	4.2	268	226	128	134	84	57	59
Teflon (TFE)	9	4.7	606	465	153	406	77	33	88
AF-E-124D	22	4.8	352	297	177	266	85	60	90

*All data except AF-E-124D from report No. 14771-6001-R0-00.

At ambient temperature a load is applied to the center of the diaphragm until approximately 0.37 inches deflection is obtained. The Dewar is then filled with LN_2 and the load is recorded at this deflection until the temperature stabilizes at approximately -320°F. The sample is then warmed to room temperature and load-deflection data again taken. These data are shown in Table 5-3 for AF-E-124D.

For Teflon TFE the load deformation relationship is as much a function of time and temperature as load. Figure 5-6 plots time, load and temperature indicating the set characteristics after load is applied. After reaching room temperature equilibrium following the LN_2 cycle, a room temperature load deflection test was run to establish properties after a cryogenic cycle.

Table 5-2. Flexure Test Data

Material	Deflection (inches)	Load (lb)	Temperature (°F)
AF-E-124D (Sample Dim 1-1/2" Dia x 0.087 thick)	0	0	70
	0.2289	3.13	70
	0.3701	6.17	70
	0.375	47.7	-320
	0.3769	6.7	70
	0.3547	3.13	70
	0.010 - 0.020	0	70

5.1.2 Flexure Tests

This test was not run during the Phase I effort and was included in this program to obtain more characteristics data on AF-E-124D since there is little property data. The load deflection characteristics of thin membrane sections of AF-E-124D material were determined, with Teflon TFE used as a control sample. Although not directly applicable to seal design and performance, a good measurement of flexibility and strength of thin sections is provided by this test.

The test set-up and fixture are shown schematically in Figure 5-4. As shown in this figure, the test equipment and set-up are essentially identical to that used in the compression tests with the exception of the sample and fixture. The test samples are in the form of diaphragms 1-1/2 inch in diameter. The samples are firmly clamped around the edges by a clamping ring as shown in Figure 5-5.

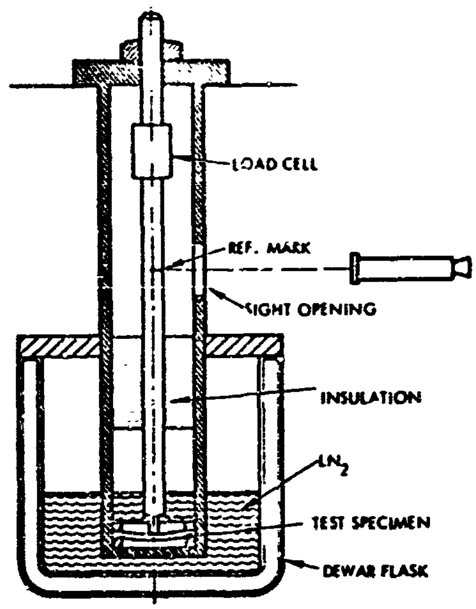


Figure 5-4. Flexure Test Schematic

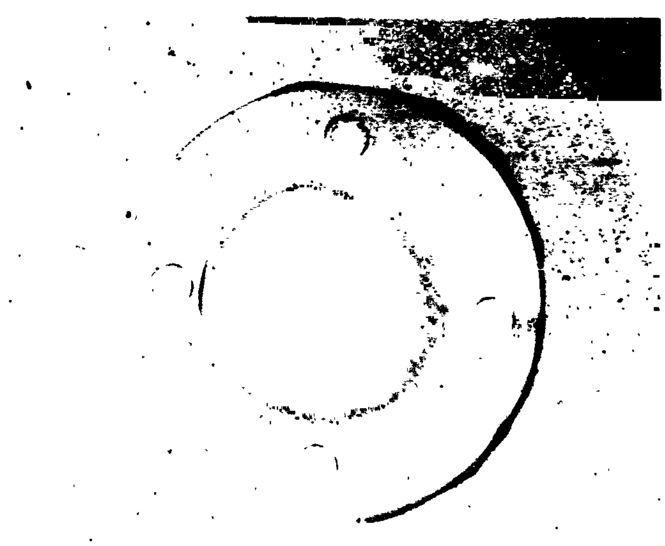


Figure 5-5. Flexure Test Clamping Device

Photographs of two samples (one AF-E-124D and one Teflon TFE) after test are shown in Figures 5-7 through 5-9. In Figures 5-7 and 5-8 the permanent set of the Teflon sample after the single test cycle is shown. The side view shown in Figure 5-7 illustrates the degree of set, Figure 5-8 shows the "worst case" AF-E-124D sample. Permanent set was seen in this sample which was not seen in a previous test. The same AF-E-124D test sample was used for two test cycles since no permanent deformation had been observed from the first test. The explanation for the difference in permanent set characteristics was that the applied load at -320°F, was 25.6 lb. in the first set and 47.7 lb. in the second, locally overstressing the material.

5.1.3 Valve Seal Tests

This test series was conducted identically with the LN_2 tests in Phase I,[1] with the exception that a minimum of 100 cycles were run after determining the initial seal load to effect seal lakage, rather than the six cycles previously run. This is considered to be more likely to develop an indication of improvement or degradation as a function of cycling.

The valve shown schematically in Figure 5-10, with the test seal material installed, was installed in a Dewar and submerged in LN_2. After temperature stabilization, helium pressure was applied to the valve inlet and verification of flow was obtained. Leakage was determined by the water displacement method, proven earlier to be an accurate and consistent method of leakage determination even when testing with liquid hydrogen. Helium pressure was then applied to the dome actuation pressure port and gradually increased until the flow stopped. The valve inlet pressure was 400 psig. During the cool-down periods, the valve was left in the open position to preclude the possibility of the seal conforming to the seat at room temperature and subsequently exactly mating with the seat after hardening at cryogenic temperatures.

After determination of the initial seal loading, the valve was cycled an additional 100 times. The actuator dome pressure was then gradually increased until flow through the valve stopped.

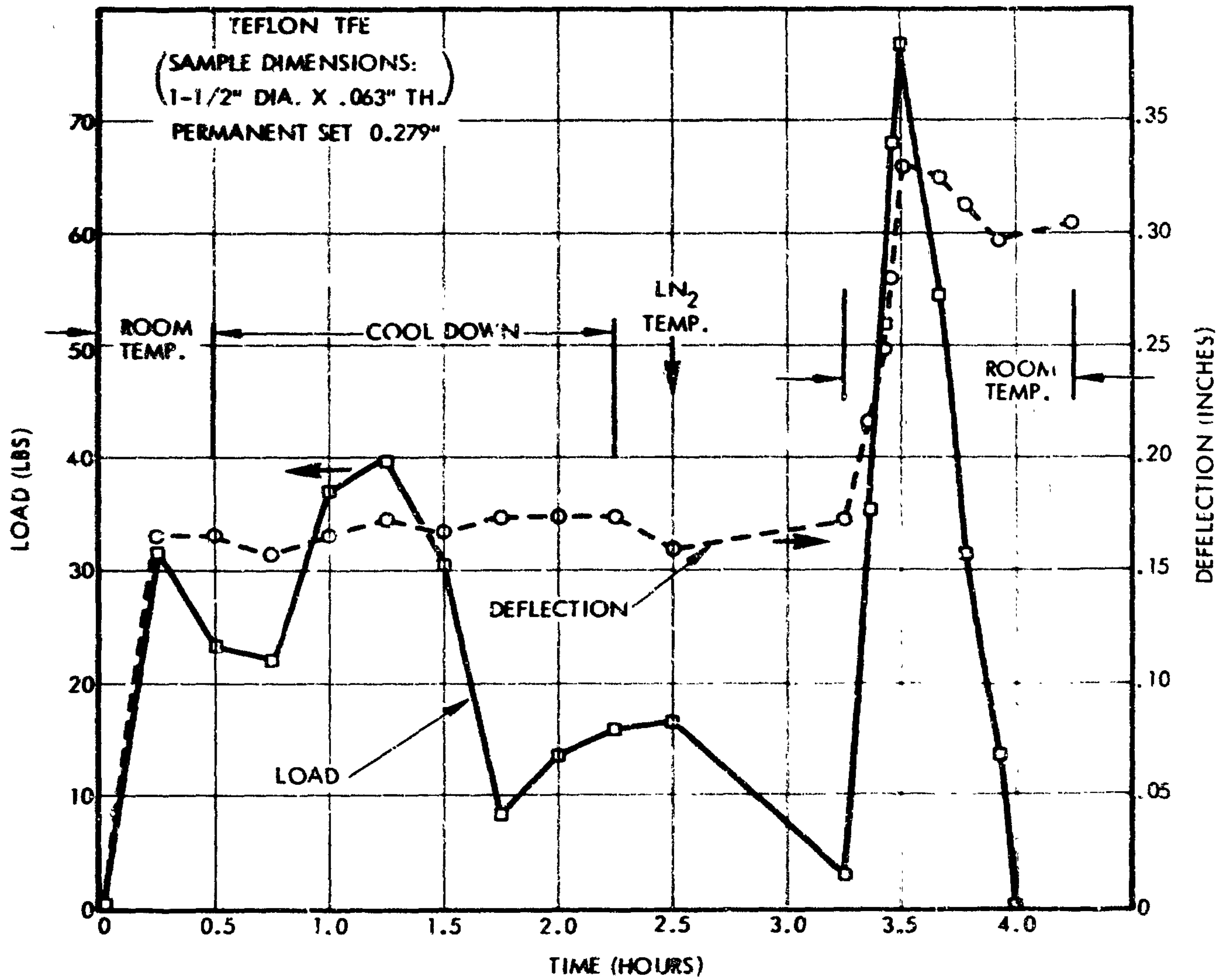


Figure 5-6. Teflon Flexure Test Data

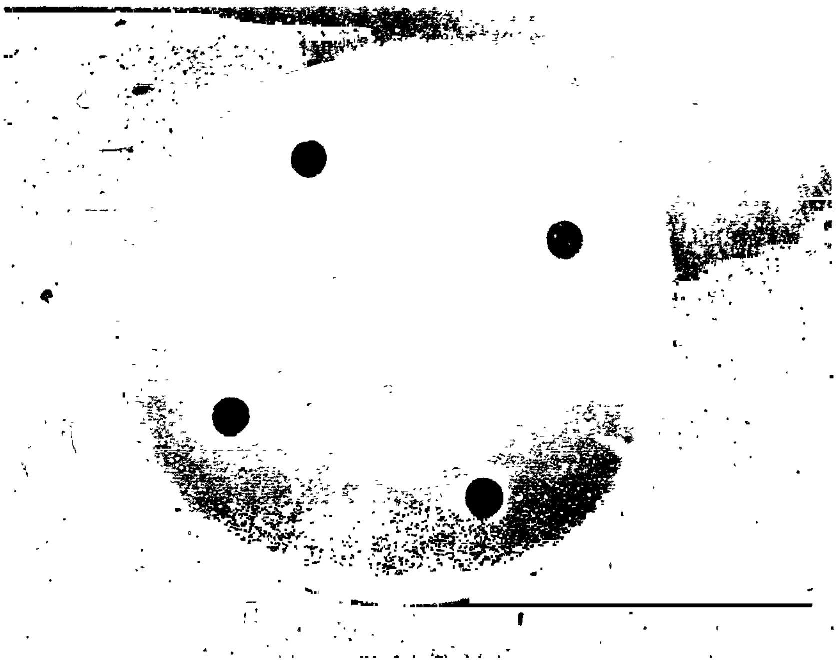


Figure 5-7. Teflon Flexure Test Specimen, Top View

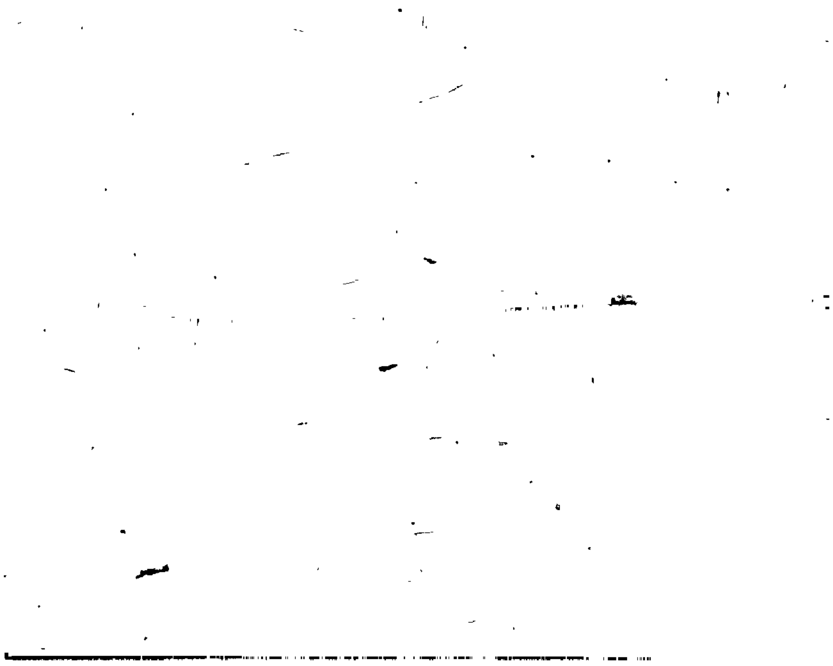


Figure 5-8. Teflon Flexure Test Specimen, Side View

Figure 5-9. AF-E-124D Flexure Test Specimen
Side View

The test seals had a surface finish of 8 to 16 microinches as molded. Considerable care was taken during preparation and molding to ensure a smooth finish on the AF-E-124D material, to obtain better finishes than were available during Phase I. The valve seat finish was also the same as previously used, a 32 microinch finish.

The test results are summarized in Table 5-3, while the detailed test data are provided in the appendices. These data reflect the relationship seen earlier, wherein elastomers seal at a lower seat stress than Teflon. The relationship at -320°F of 32 percent lower seat stress to seal AF-E-124D than Teflon compares favorably with the LN_2 data from Phase I where the difference was 39 percent. The seal condition after the tests are shown in Figures 5-11 and 5-12. The degree of compression set is comparable with previous results and indicates good recovery of the AF-E-124D.

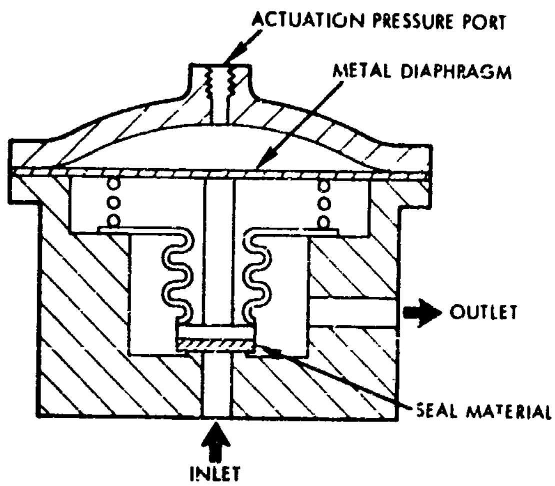


Figure 5-10. Schematic Poppet Seal Test Fixture

5.2 SOLVENT EXPOSURE TESTS

Each material (AF-E-124D and Teflon) was subjected to a series of solvent exposure tests to establish fluid compatibility. Although the Teflon material was tested during Phase I, samples were again run in parallel with each AF-E-124D sample to serve as a control. The fluids used in this test are common cleaning and test fluids normally used in propellant systems. They are:

- Distilled water
- Freon TF
- Isopropyl Alcohol (IPA)
- Trichlorethylene

The test procedure is as follows: First, samples of standard sizes were prepared, (Tensile specimens approximately 1.5 inches long, 0.060-0.090 thick and 0.19 wide in center). Measurements of length, thickness width and weight were recorded. The samples were placed in individual containers with each fluid, and exposed to the fluids for the following lengths of time:

- One hour
- Four hours
- One day
- Three days
- Seven days
- Fourteen days

Table 5-3. Summary of LN_2 Valve Seal Test Results

Material	Valve GHe Inlet Pressure psi	Seat Stress to Effect Zero Leakage, psi	Temperature °F
AF-E-124D	400	690	+ 70
	400	2530	-320
(After 100 cycles)	400	2500	-320
	400	860	+ 70
Teflon TFE	400	1260	+ 70
	400	3740	-320
After 100 cycles	400	3570	-320
	400	1210	+ 70

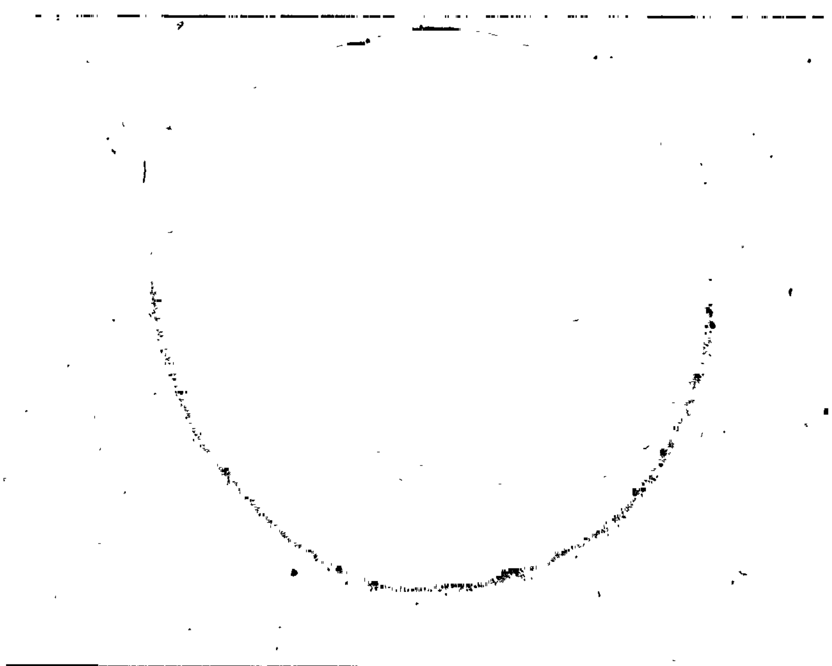


Figure 5-11. Teflon Valve Seal After LN_2 Test (10X)

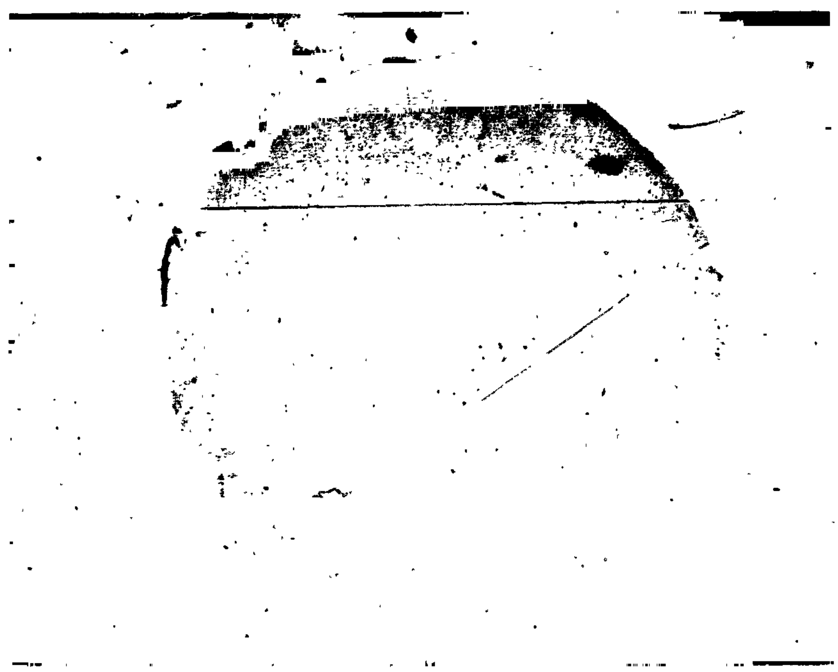


Figure 5-12. AF-E-124D Valve Seal After LN_2 Test (10X)

At the end of each exposure period the samples were removed from the solvent, wiped dry and immediately weighed and remeasured. The samples were then air dried for approximately one week, at the end of which time they were again reweighed and remeasured. Then each sample was tensile tested to determine the ultimate strength and the elongation of the sample at rupture.

Test results based on a very limited number of tests were reported in the Phase I report. The additional tests with AF-E-124D confirm those results which indicate generally;

Distilled Water	-	Compatible, no appreciable change
Freon TF	-	Exhibits some incompatibility - not recommended
Isopropyl Alcohol	-	Compatible
Trichlorethylene	-	Exhibits some incompatibility - not recommended

Table 5-4 indicates a summary of data for both the AF-E-124D and Teflon. More detailed information is provided in the appendices.

Table 5-4. Summary AF-E-124D and Teflon TFE Solvent Exposure Results

Solvent	Exposure Time	AF-E-124D PERCENT CHANGE					Teflon TFE PERCENT CHANGE				
		Dim.	Wt.	Load at 100% Elong	Elong at Rupture	Tensile	Dim.	Wt.	Load at 100% Elong	Elong at Rupture	Tensile
Distilled Water	Short term (1 day)	0	+ .01	+ 2.4	+ 5.2	0	0	0	+23	0	+10.7
	Long term (14 days)	0	+ .05	+ 5.2	+12.0	0	+3.3	0	+21	0	+10.7
Freon TF	Short term	+20	+40	-47.5	+ 5.2	-70.0	+2.6	+0.7	+20.0	+ 5.2	+ 5.2
	Long term	+16	+37	-56.5	+12.0	-60.0	+3.1	+1.1	+ 9.9	+10.9	+ 9.1
IPA	Short term	+ .1	0	+ 5.2	+ 5.2	+14.7	+4 8	0	+10	+ 5.2	+ 7.5
	Long term	+ .1	0	0	+13.2	+14.7	+2.3	0	+21	-21.2	+ 4.7
Trichloroethylene	Short term	+10	+ .4	0	+ 9.5	+22.0	0	+ .2	+16	-21	+ 5.3
	Long term	+ 1	[illegible] 1.2	- 6.0	+ 7.9	-17.6	+4.1	+1.5	+16	-21	+12.1

5.3 Compression Set Tests - Ambient and Elevated Temperature

Compression set, or cold flow is one of the most limiting characteristics of Teflon in terms of seal design. In view of similarities between Teflon and AF-E-124D it was considered important for purposes of this program to make measurements of the compression set of AF-E-124D as well as comparative measurements on Teflon.

Test specimens consisted of 0.5 inch diameter discs cut from sheet stock and plied to provide a thickness of approximately one-half the diameter. The compression set fixture is shown in Figure 5-13. The initial compression, or squeeze ranged between 22 and 30% for the AF-E-124D samples and between 12 and 17% for the Teflon samples. Compressed samples were stored in air at temperatures ranging from room temperature to +200°F, for time periods of 24 and 100 hours. The compression set was measured at various times after the samples were removed from the fixture. It is interesting to note that whereas most rubbers will reach an equilibrium recovery after one half hour, AF-E-124D recovered significantly up to one hour after unloading following the room temperature test. The data for AF-E-124D and Teflon is shown respectively in Tables 5-5 and 5-6. After 24 hours at 77°F the AF-E-124D sample had a 14.1% set after one hour recovery, whereas under the same conditions the Teflon sample had a 68% set. At 200°F for 24 hours the Teflon sample set 95% in comparison with 71% for the AF-E-124D sample. Typical values for comparison set for most elastomers at 160°F for 24 hours range between 15 to 30%. As seen from Table 5-5 the value measured for AF-E-124D was 68.5%. It is important to note that whereas the standard ASTM compression set specimen is 0.5 inch thick, the data shown in Tables 5-5 and 5-6 are for 0.25 inch thick specimens. The test conditions are more severe for the thinner specimen. This should be taken into account in comparing this data with other test data.

A single compression set test conducted per ASTM-D-395, Method B, using a one inch diameter by 1/2 inch thick AF-E-124D test specimen resulted in a significantly lower compression set value than obtained with the 1/2 inch discs. A 22 hour test at 300°F resulted in a compression set of 41%. Comparison of data in this report should therefore be limited to specimens of equal configuration.

An important conclusion which can be drawn from Tables 5-5 and 5-6 is the superiority of AF-E-124D over Teflon in terms of recovery after being loaded, or the elastic memory of the material.

It should be noted that these compression set values are computed on the basis of percent of original deflection as opposed to percent of original thickness, the method used elsewhere in this report.

The method used in Tables 5-5 and 5-6 is consistent with ASTM techniques for measuring comparison set. It is also significant to note in considering the compression set characteristics of AF-E-124D that there has been very little evidence of compression set based on valve seat tests conducted to date as indicated by leakage after cycling and visual examination of seals.

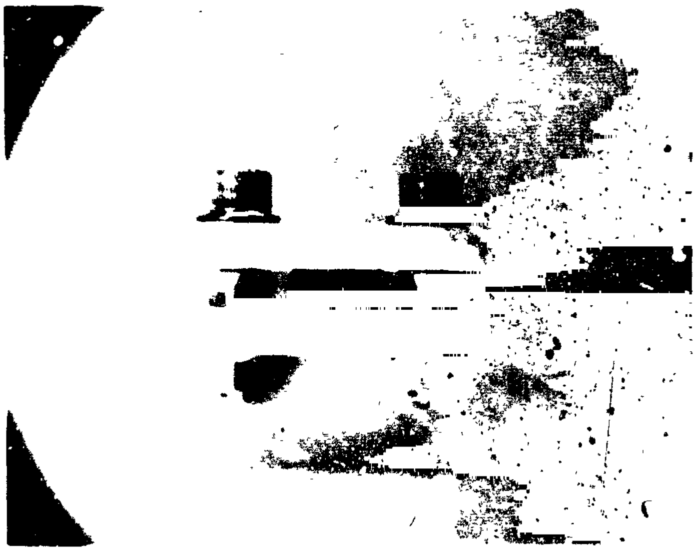


Figure 5-13. Compression Set Fixture

Table 5-5* Compression Set of AF-E-124D after 22 to 30% compression

$$(\% \text{ Set} = \frac{\text{Initial Thickness-Final Thickness}}{\text{Initial Thickness-Compressed Thickness}})$$

Time, Hr	Temperature, °F	Initial Compression, %	Compression Set, %			
			After 1/2 Hr	After 1 Hr	After 2 Hr	After 80 Hr
24	77	29.3	19.2	14.1	14.1	2.6
24	160	22.5	68.5	66.5	66.5	58.5
24	200	27.0	71.0	71.0	71.0	61.0
100	77	23.2	36.5	35.5	35.5	11.8
100	200	26.7	79.0	78.0	78.0	67.0

NOTE: Values are not comparable to ASTM test data because of specimen configuration.

Table 5-6* Compression Set of Teflon after 10 to 14% compression

$$(\% \text{ Set} = \frac{\text{Initial Thickness-Final Thickness}}{\text{Initial Thickness-Compressed Thickness}})$$

Time, Hr	Temperature, °F	Initial Compression, %	Compression Set, %			
			After 1/2 Hr	After 1 Hr	After 2 Hr	After 80 Hr
24	77	10.4	78	78	-	66.5
24	200	9.7	98	98	98	98
175	77	10.2	85	85	85	85
100	200	14.3	98.5	98.5	98.5	96

*Specimen configuration: 0.5 inch diameter X .25 inch high

6. COMPARISON AND CORRELATION OF AF-E-124D DATA

The information obtained during this program, in general serves to substantiate the potential capability of AF-E-124D as an advanced seal material for a wide range of applications. Two methods of comparison were used; a quantitative comparison in all areas possible, using tabular data from the Phase I report; and a qualitative comparison, discussing the properties of AF-E-124D, as compared, to other materials, using TRW generated data and other sources. This information is presented here to provide as much data as possible on the characteristics of AF-E-124D material. There is conflicting information from these various sources as discussed later, but recognizing the developmental status of the material at this point, this is not surprising.

6.1 COMPARISON WITH PREVIOUS DATA

Table 6-1, presents the summation and conclusions from Phase I, modified to reflect the LN_2 data from this program. Also included is the permanent set characteristics at ambient temperature and 200°F after 24 hour and 100 hour exposure periods.

These data show a 14% room temperature set for AF-E-124D compared to approximately 70% Teflon permanent set. Elevated temperature data, however, indicate a greater permanent set than expected from the retention of properties up to temperatures as high as +400°F obtained in Phase I. As indicated earlier the test technique will influence the results of this type of test. The design of the seal will influence the degree of comparison set, e.g. a thin seal will exhibit a higher percentage permanent set at elevated temperature than a relatively thick seal.

Table 6-1. Summary of Seal Material Performance

Rating			Material	Sealing Capability Ratio of Load Required to Seal at Cryogenic Temperature to Ambient Temperature		LO_2 Impact Resistance Maximum Impact with no Reaction ft-lbs	Resistance to Permanent Seat Deformation (3)	High Temperature Resistance, Average Percent Change in Tensile Strength		Permanent Set after 24 hrs Compression %	
H_2	O_2	O_2/H_2		LH_2	LN_2 (Revised)		+60 to -423°F	+400°F	+250°F	+77°	+290°F
2	1	1	AF-E-124D	6.0	6.5 (2.9)	72	Excellent	-20	-27	14[4]	71[5]
4	2	2	Teflon TFE	10.0	3.7 (3.1)	72	Fair	0	0	78[5]	95[7]
3	6	6	255-2 (Viton A)	5.0	4.5	40	Good	0	-12		
8	7	7	310-1 (Phenyl Silicone)	4.7	3.4	<20	Good	-26	-14		
1	***	***	HYSTL	3.7	2.9	<10	Excellent	0	0		
**	***	***	316-1 (Fluorosilicone)	**	12.5	<10	Poor	-50	-28		
12	***	***	AF-E-71-2	11.6	6.	<10	Fair	-93	-54		
11	***	***	263-2 (EPT-HYDRIN)	6.3	(1)	<10	Fair	*	0		
9	***	***	Mylar (2)								
5	3	3	Kel-F (2)								
16	***	***	Nylon (2)								
7	5	5	Kynar (2)								
6	4	4	Vespel (2)								

*Material Decomposed
**Material failed under load.
***Not rated due to LO_2 impact sensitivity
(1) Not tested.
(2) Not tested during this program.
(3) Definition of terms:
Excellent - No evidence of permanent set
Good - Slight seat imprint
Fair - Medium to deep seat imprint
Poor - Seat cracked
(4) 29% initial compression
(5) 27% initial compression
(6) 10.4% initial compression
(7) 9.7% initial compression

Table 5-1 (shown in Section 5) indicates that at cryogenic temperatures AF-E-124D compares favorably with other materials tested previously. This data also indicates one area of weakness, after a cryogenic cycle, that of permanent set, initially approximately equivalent to Teflon. Although rapid recovery occurs to approximately one third the value of Teflon, or equal to other elastomers. In all other respects, the material exhibits the desirable properties required of an improved seal material. Room temperature set is approximately 25% that of Teflon, and 200°F compression set is 40 to 70% that of Teflon. Typical room temperature compression set of elastomers is in the range of 10 to 20%, and elevated temperature (160°F) compression set is approximately 30%.

6.2 RELATED AF-E-124D DATA

The following is a summary of related work on AF-E-124D being conducted by NASA and the Air Force.

6.2.1 Related Jet Propulsion Laboratory (JPL) Seal Materials Evaluation

JPL is conducting in-house compounding, materials tests, and valve tests as well as contracted work involving the perfluorinated elastomers. Specific compound designations being evaluated by JPL are LRV 448, ECD-006 and AF-E-124D. To date testing of AF-E-124D has been limited to evaluating the sealing characteristics of the material in a cycle test fixture.[6] Seal test specimens consisted of discs cut from sheet material provided to JPL by TRW. Table 6-2 summarizes room temperature leakage data. Leakage was measured after the number of cycles indicated. The AF-E-124D material maintained the lowest leakage. The test valve which had a 0.050 ID seat is illustrated in Figure 6-1. The seal is independently spring loaded to accommodate thermal growth at elevated temperatures. The seal compression was limited by a metal to metal positive stop in the plunger to 0.0025 inches, and the seal spring provided a 0.6 lb load. GN_2 leak tests were run at 60 psig during cycling. The pressure load plus spring load resulted in a sealing stress of approximately 700 psi. The same seat stress was used for all seal materials.

Visual comparison after testing showed that the ECD-006 material took a greater set than AF-E-124D. The low leakage and the seat

Table 6-2. Valve Seat Leakage Data-Room Temperature Cycles (Data from Ref. 6)

Seal Material	No. Cycles	GN_2 Leakage at 60 psig (scc/hr)
Kel-F 81	25,000	10
Grade 7 TFE	50,000	0.5
AF-E-102	1,000,000	1.2
TFE Glass Filled	25,000	0.4
TFE Graphite Filled	25,000	1.5
ECD-006	10,000	1.2
AF-E-124D	950,000	0.16

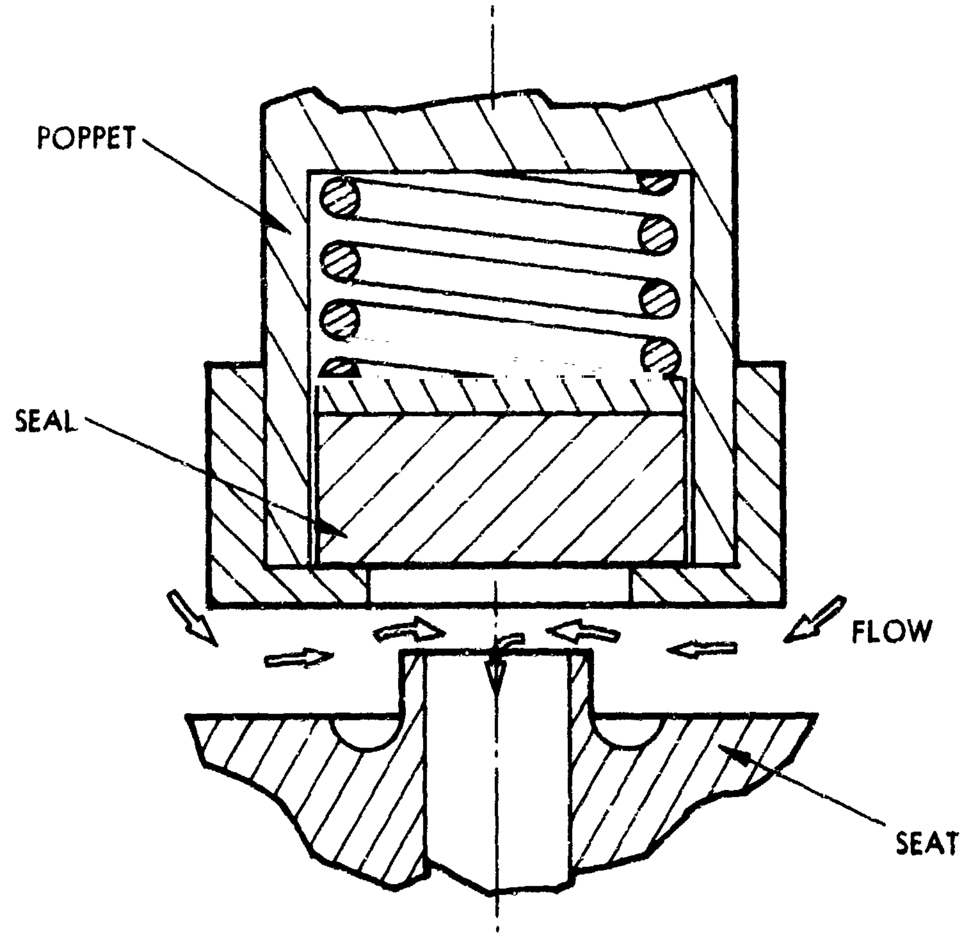


Figure 6-1. Schematic of JPL Seal Test Configuration

penetration of 0.0025 inches illustrate that the AF-E-124D after approximately 10^6 cycles took very little compression set. During this same test series the ECD-006 seat was subjected to 10,000 cycles at -70°F. The leakage was found to be excessive. Based on measuring leakage as a function of progressively reducing temperature from +390°F when the leakage was zero it was determined that a lower temperature limit for ECD-006 was +20°F based on an allowable leakage of 1.0 scc/hr. Low temperature tests were not run on the AF-E-124D material. (Leakage tests run by THW during Phase I however demonstrated the ability of AF-E-124D to provide a satisfactory seal at temperatures as low as -423°F.[1]) Although both materials are considered to be chemically similar the conclusions drawn relative to both ambient and lower temperature sealing performance of perfluorinated elastomers based on these two tests are obviously different. The results indicate that the AF-E-124D material performs better as a seal than ECD-006 both at ambient and low temperatures, the differences probably being a result of compounding or processing.

In addition to ambient and low temperature cycle tests with ECD-006, the JPL test program included cycling at +390°F. The ECD-006 seal exhibited zero GN_2 leakage after 10,000 cycles at 390°F and retained zero leakage after cooling to room temperature. This test indicates the potential of the perfluorinated elastomers for elevated temperature service.

6.2.2 Related Work by TRW Under Contract to the Air Force Materials Laboratory (AFML)

Under a program entitled "Development of Elastomeric and Compliant Materials Resistant to Liquid Rocket Propellants," (Contract No. F33615-70-C-1514),[3] [5] TRW, under the direction of AFML is evaluating the compatibility of perfluorinated elastomers with various propellants. Three different material samples were provided by AFML to TRW for test and evaluation. The three materials were designated ML-124D, ML-137, and ML-144. Propellants to which the materials have been exposed include Hydrazine (N_2H_4), nitrogen tetroxide (N_2O_4), and chlorine trifluoride (ClF_3) at temperatures ranging from room temperature to +165°F.

Based on retention of mechanical properties the AF-E-124D compound was superior in all tests performed. The data for ClF_3 and N_2H_4 are shown in Table 6-3. Data for exposure to N_2O_4 are shown in Tables 6-4 and 6-5. It will be noted that the mechanical properties of the control samples in all three tests were different, i.e., Shore A hardness ranged from 69 to 80. This fact points to variations in processing. The handling and curing procedures for these materials are far more complex than for typical elastomers.

Variations in the data points to the need for developing processing procedures and controls for achieving optimum properties and maintaining batch to batch consistency.

The excellent retention of mechanical properties after propellant exposure indicates the potential of perfluorinated elastomers as not only

Table 6-3. Effect of 8-Day Propellant Exposure on AF-E-124D Properties

Property	Control, Prepropellant Immersion		CTF Ambient Temp.		N_2H_4 140°F	
	Avg.	Std. Dev.	Avg.	Std. Dev.	Avg.	Std. Dev.
Hardness, Shore A	80	—	80	—	77	—
Tensile Strength[a] at Break, psi	2580	200	2380	400	2250	250
Elongation[a] at Break, %	160	6	160	6	150	12
Modulus[a] at 100% psi	890	105	880	55	1050	45
Weight Change, %	—	—	+2.0[b]	0.0	+1.7	0.1
Length Change, %	—	—	0	0	0	0
Tensile Set, %	8	0	6	0	0	0
Appearance	Colorless, translucent		Colorless, translucent		Brown, translucent	

[a]Tested at 72°F with a crosshead speed of 20 inch/min.

[b]Weighed after 77 hours vacuum pumping

Table 6-4. Effect of 8-Day Nitrogen Tetroxide Exposure on AF-E-124D Properties

Property	Control Mean ± Std. Dev.	≈72°F Propellant Mean ± Std. Dev.	120°F Propellant Mean ± Std. Dev.
Shore A Hardness	69	71	69
Tensile Strength, psi	2335 ± 120	2300 ± 180	1500 - 2450[a)]
Modulus at 100% Elongation, psi	910 ± 45	770 ± 20	745 ± 75
Elongation at Break, %	155 ± 7	155 ± 11	145 ± 21
Tensile Set, %	3 ± 0	3 ± 0	0 ± 0

[a]Erratic results

Table 6-5. Effect of 8-Day 165° Nitrogen Tetroxide Exposure on AF-E-124D Properties

Property	AF-E-124D	
	Control (Unexposed)	Exposed
Shore A Hardness	78	70
Tensile Strength, psi	2600	1973
Modulus at 100% Elongation, psi	835	517
Elongation at Break, %	150	167
Tensile Set, %	2	4
Volume Swelling, %	—	16
Change in Weight, %	—	12

good low temperature and high temperature seal materials but also for broad applications with earth storable fuels and oxidizers including fluorinated oxidizers. Although limited, the data generated to date with the perfluorinated elastomers indicate the potential for a universal seal material. Of the compounds for which data are available AF-E-124D has demonstrated the best combination of characteristics.

6.3 COMPARISON OF AF-E-124D PROPERTIES AND CHARACTERISTICS

Characteristics of AF-E-124D as they compare with other seal materials are discussed in the following paragraphs.

6.3.1 Mechanical Properties

Although compression is more important in most seal applications than tension tensile strength, it is an important indicator of mechanical properties and for seal applications, 2000 psi is considered to be a good nominal strength value. Most elastomers exceed this value, exceptions being polysulfide and silicone which typically have tensile strengths in the order of 1500 psi. As seen from Table 6-4, AF-E-124D has a tensile strength in excess of 2000 psi and even after exposure to N_2O_4 for eight days at 165°F it retained a strength of 1975 psi.

Hardness is an important measure of the ease with which a material can effect a seal. The optimum hardness for elastomer seals is in the 70-90 Shore A range. All plastic materials with the exception of polyvinyl chloride exceed this hardness range. The hardness of Teflon measured on a Shore A scale exceeds 95. AF-E-124D with a hardness in the range of 70-80 Shore A is ideal for sealing, requiring only a minimum concern for surface finishes or contamination limits.

Percent elongation gives an indication of a material's compliance and resilience, or ability to conform to a mating surface and maintain a sealing load. Typically, materials with less than 100% elongation require accurate surfaces to account for lack of resilience and compliance. Such seal materials include nylon, Kel-F, mylar, polyimides (Vespel) and acetal (Delrin). At an elongation in excess of 150%, AF-E-124D has a low elongation compared with typical elastomers (200-400%), however it is adequate to provide good sealing without requiring accurate surface finishes.

6.3.2 Propellant Compatibility

Taking into consideration the limited amount of testing and the fact that compatibility results are significantly influenced by compounding and processing variations, the perfluorinated elastomers appear to be compatible with a wide range of fluids. Resistance to oxidizers was demonstrated in LO_2 impact tests during the Phase 1 program. Of the plastics and elastomers tested, which included in addition to AF-E-124D, Teflon, Viton, phenyl silicone, fluoro silicone and ethylene propylene rubber, only Teflon and AF-E-124D showed no reactivity with LO_2.

Changes in hardness and volume swell are often considered key indicators of compatibility for materials to be used as liquid propellant seals. As indicated in Table 6-3, there was no change in hardness and no length change (volume swell) after AF-E-124D was exposed to ClF_3 for eight days. After eight days in 140°F N_2H_4 the material lost only 3 points of hardness (a change of $\pm$ 5 points is commonly considered acceptable) and evidenced no dimensional change. The measured change of 2 points in hardness after exposure to N_2O_4 at 72°F and 120°F for eight days (Table 6-4) is considered to be within experimental error.

Although these data are not conclusive they indicate that AF-E-124D is unique among elastomers in propellant compatibility comparing favorably with Teflon, which stands alone among polymers in terms of its chemical inertness.

6.3.3 Thermal Resistance

One of the most unusual characteristics of the perfluoroelastomers is the wide temperature range over which the material appears to be serviceable for seal applications. In the JPL work, testing with EDC-006 demonstrated satisfactorily seal performance at temperatures up to +390°F, (which was the highest test temperature). The only elastomers generally recommended for service temperatures of +400°F and above are the silicones and fluoroelastomers (Viton and Kel-F elastomer). The TRW work on this program has demonstrated the applicability of AF-E-124D as a seal at -423°F. In these seal tests, reported in the Final Report Phase I,[1] with the exception of HYSTL, AF-E-124D was the only material tested which showed no physical change (seat impression or cracking) after being tested at -423°F. At -423°F a fluorosilicone material consistently cracked and Teflon required over 60% greater sealing load to effect a seal than AF-E-124D, and retained a deep permanent seat impression following the test.

6.3.4 Gum Strength

The strength of the pure unfilled gum stock is low for many synthetic rubbers and additives are typically required to develop adequate strength, whereas the perfluorinated elastomers achieve their physical properties without the need for fillers, plasticizers, antioxidants, etc. Carbon black is one of the most common fillers added to rubbers, and antioxidants are added to inhibit oxidation. Additives are often a prime limiting factor in terms of propellant compatibility. Sulfur, commonly found in carbon black for example can cause N_2H_4 decomposition. The leaching out of plasticizer causes certain elastomers to become harder and less resilient after exposure to propellants.

By not requiring additives AF-E-124D minimizes the problem of maintaining consistent quality due to variations in quantity and purity of ingredients. A fraction of a percent change in the amount of sulfur in ethylenpropylene rubber has been found to significantly affect the degree of change in physical properties after exposure to N_2H_4. Other fluorinated elastomers including Viton, Kel-F elastomer and fluorosilicone are compounded with fillers and plasticizers to develop satisfactory mechanical properties. The fact that the perfluorinated elastomers can be used as neat polymers is in itself a very desirable feature in terms of material quality.

7. SEAL DESIGN CONSIDERATIONS

It is apparent from the results of the seal materials development program that the most critical design problem to be faced in actual applications will be the extremely broad range of seat loads found to be required to effect a seal at the temperature extremes of -400° and $+200^{\circ}F$. Specifically, the high loads required at cryogenic temperatures, will extrude and permanently deform elestomeric seals when the same load is applied at high temperatures. It is for this reason that hard plastic type materials have conventionally been used as seals for these applications. This practice, however, has resulted in inferior sealing characteristics at the higher temperatures and also affected endurance capabilities and tolerance to contaminants.

The identification of AF-E-124D (an elastomer) as being compatible with LO_2, having successfully met the ABMA impact requirements, further increases the desirability of developing an elastomeric seal configuration capable of tolerating a b[illegible] range of seat loads. Its high temperature stability in air is indicative of an inherent capability of being compatible with oxidizers in general, thus enhancing its potential serviceability in rocket propellant fluid control devices.

A design concept offering a solution to this problem should be basically simple in construction in order to provide long term reliability. To serve as a design guide, a list of requirements is presented below. The design must be capable of:

- Generating seal load of approximately 2500 lb/in^2 min at temperatures below the Tg of the seal material and 600 lb/in^2 max at temperatures above the softening point.
- Tolerating the relatively large differential expansion common to most elastomers.
- Fast response to temperature changes and to withstand temperature shocks without damage.
- Resisting flow erosion or nibbling, especially at the higher operating temperatures.
- Providing positive means for resisting permanent set.
- Providing close poppet guidance resulting in repeatable seat imprints and a minumum of seat scuffing.

The feasibility of reducing these relatively severe and conflicting requirements to a producible design appears to be practical. Figure 7-1 illustrates schematically the TRW "center STOP" seal design, developed for use with small attitude control thruster engines. This design, using EPT seal materials, specifically compounded for low swell in hydrazine (<2% at 200°F) was successful in solving the problem of progressive seal set with continual use which resulted in progressively changing flow rates with time. The same design concept would be equally applicable to meeting the above listed requirements by absorbing the high loads required at low temperatures by means of the stop when operating at high temperatures.

Other design approaches could incorporate bi-metal temperature compensating devices configured to retract at low temperatures and to absorb some of the load at high temperatures. Such devices are relatively simple and extremely reliable, having been similarly used for years in other applications. It is also feasible to configure the seal and seat design such that as the seal softens with increasing temperature, additional seal material gradually comes into bearing resulting in progressively reduced unit loading. A proper design will thus be self compensating and involves no moving parts. The use of an elastomer as the seal material has the advantage that after removal of load the deformation relaxes and the seal assumes its original configuration if not overstressed.

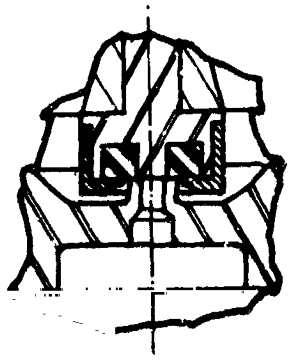


Figure 7-1. Center Stop Seal Design

To preclude leakage occurring around the back of a seal at cryogenic temperatures, a common failure mode, the elastomer may be bonded in place. TRW has exposed typical bonded EPR seals to leakage tests at LN_2 temperatures with satisfactory results. Repeated load cycle tests conducted with these seals at -300°F have not resulted in any damage to the seal or the bond. The bonding of AF-E-124D has not been attempted and would undoubtedly require the compounding of a special bonding agent. However, the design simplifications potentially available if proven bonding processes were developed are obvious. The use of bonding as a means of seal retention eliminates the multiple problems associated with mechanical forms generally used. The loosening of a seal at low temperatures due to the larger contraction of polymers as compared to metals is fundamental and virtually impossible to tolerate if operation at +200 to +400°F is also a requirement. Some of the materials (i.e., Viton) tested during Phase I for which bonding agents and procedures are available can be readily joined to various different base metals, consequently the use of this retention technique is strongly recommended for further evaluation.

8. CONCLUSIONS AND RECOMMENDATIONS

8.1 CONCLUSIONS

The tests conducted during this program, along with other available data indicate that AF-E-124D is a material which possesses characteristics desirable in a material for seals for oxygen/hydrogen propulsion systems. In addition there is evidence that this material is suitable for propulsion systems using other propellants. This program reinforced some of the findings of the earlier program under contract NAS 9-10481 indicating that AF-E-124D is a superior seal material for cryogenic seal applications.

It is important to point out that the perfluorinated elastomers are still experimental in nature, and that variations in data characterizing specific compounds should be viewed as indicative of the potential of the material when optimized in terms of compounding and processing. The processing of perfluorinated elastomers at present is very critical in the development of properties. Significant property differences have been obtained as a result of processing, such that the characteristics data obtained from other perfluorinated elastomers may not apply to AF-E-124D. The test results obtained from AF-E-124D are generally superior to those of other perfluorinated elastomers. From the data available, AF-E-124D as a seal material has shown the following characteristics:

- Effective sealing at cryogenic temperatures, at a lower seat stress than required with Teflon
- Ability to deform at -320°F without appreciable permanent set
- Generally superior sealing ability and lower set characteristics than other perfluorinated elastomers
- Demonstrated compatibility with LO_2 and LH_2, as well as N_2O_4 N_2H_4 and ClF_3 indicating the possibility of a near universal propellant seal material
- May be a satisfactory valve seal material up to 400°F (based on test data of ECD-006, another perfluorinated elastomer)
- Although seal tests have not shown compression set to be a problem, standard compression set test data indicate that this is the weakest mechanical property, particularly at elevated temperature.

Table 8-1 presents summary data provided in contract NAS 9-10481 with the results of this program added. The property data are approximately equal with the exception that Teflon is compatible with more cleaning fluids, while AF-E-124D exhibits no observable compression set in valve tests at ambient temperature and no degradation during low temperature testing.

8.2 RECOMMENDATIONS

The primary recommendation resulting from this program is that AF-E-124D be applied to actual seal designs and subjected to testing. As with any seal material, designs must be developed which take advantage of the material strengths and minimize effects of the weaknesses.

Recommendations for continuing effort include:

- Continue characterization of AF-E-124D material and compounds, which includes controlled optimized processing, with established variables and tolerances
- Design, fabricate and test valve seal designs which provide increasing load with decreasing temperature.
- Conduct valve and static seal tests with AF-E-124D Viton-A, Viton EC-60 (an improved compound) HYSTL and Teflon TFE, simulating "in service" conditions with LH_2 and LO_2.
- Establish the optimum seal load for AF-E-124D
- Investigate methods of compounding AF-E-124D to reduce compression set.

Table 8-1. Comparison of AF-E-124D and Teflon TFE

Mechanical Properties

	AF-E-124D		Teflon TFE	
	Initial or Ambient	Post Test	Initial or Ambient	Post Test
1. Tensile Strength after Exposure to:				
$250^{\circ}F$	2500 psi	1812	2930 psi	2915
$325^{\circ}F$	2500 psi	1730	2930 psi	3080
$400^{\circ}F$	2500 psi	2000	2930 psi	2900
	(Current Program)		(Current Program) *	
Distilled Water	2125	2125	2185	2404
Freon TF	2125	625	2185	2290
Trichlorethylene	2125	2600	2185	2300
Isopropyl Alcohol	2125	2500	2185	2350
2. Seat Stress to Seal at 400 psi				
LH_2 Temperature	630 psi	5200 psi 2320 psi	578 psi	6900 psi
LN_2 Temperature	700 psi	3750 psi	1260 psi	3600 psi
3. Compression Set after LH_2 Valve Seal Tests	None	None	None (initial)	Extreme
4. Hardness	78 Shore A	(1)	Rockwell 58R	(1)
5. Modulus of Elasticity	835	(1)	580,000	(1)
6.* Compression Set after LN_2 Valve Seal Tests	None	None	None	Extreme

(1) Not Determined

*Indicates data from this program - other data obtained from Contract NAS 9-14081.

REFERENCES

1. H. W. Wright, "Seal Material Development Programs - Final Report, Phase I," Contract NAS 9-10481, Report No. 14771-6001-R0-00, 31 December 1970.

2. Barney, A. L., W. J. Keller and N. M. von Gulick, "A High-Performance Fluorocarbon Elastomer," J. Polymer Sci. A-1, 8, 1091 (1970).

3. Jones, R. J., J. W. Martin and P. Tarrant, "Development of Elastomeric and Compliant Materials Resistant to Liquid Rocket Propellants," First Technical Management Report under Contract F33615-70-C-1514, 15 November 1970, Page 7.

4. Personal communication J. W. Martin (TRW Systems) and J. K. Sieron (AFML).

5. Martin, J. W., "Development of Elastomers for Liquid Propellant Containment," First Semi-Annual Technical Management Report under Contract F33615-70-C-1233, 15 July 1971, Page 23.

6. "Design Support Testing and Materials Evaluation Testing of Valve Seat for 0.050 Diameter Orifice Valve," Progress Report, 18 March 1971 to 27 May 1971, Marquardt Co., Contract NAS 1-9601.

APPENDIX

LN_2 LOAD COMPRESSION TEST DATA AF-E-124D

Sample Description	Compression Inches	% Comp.	Load Pounds	Temperature °F
AF-E-124D	.0549	22.3	352	+ 70
Initial thickness .247 Final thickness .235	.056	22.7	297	+ 70 RT Equil.
Diameter .939	.061	24.8	177	-320 Equil.
	.061	24.8	266	+ 70
	.052	20.4	147	+ 70
	.041	16.5	82	+ 70
	.011	4.5	0	+ 70
(after 5 min.)	.004	1.7	0	
Teflon TFE	.022	9.1	582	+ 70
Initial thickness .237 Final thickness .225	.021	8.9	528	+ 70 RT Equil.
Diameter .935	.023	9.5	170	-320 Equil.
	.021	8.9	425	+ 70
	.020	8.7	300	+ 70
	.017	7.0	71	+ 70
	.012	5.0	0	+ 70

LN_2 FLEXURE TEST DATA

	DEFLECTION IN.	LOAD LBS.	TEMPERATURE °F		ELAPSED TIME MIN.
AF-E-124D Specimen 1-1/2" Dia. X .087" Thick	0	0	+ 70		N.A.
	.2289	3.13	+ 70		"
	.3701	6.17	+ 70		"
	.375	47.7	-320		"
	.3769	6.7	+ 70		"
	.3547	3.13	+ 70		"
	.010 - .020	0	+ 70		"
AF-E-124D	.110	25.6	-320		N.A.
	.110	7.0	+ 68		"
	.054	0	+ 68		"
TEFLON TFE Specimen 1-1/2 Dia. X .064" Thick	0	0	+ 75		0
	.0211	3.13	+ 75		
	.1648	31.73	+ 75		15
	.1659	23.03	+ 75		30
	.1573	21.93	+ 75		45
	.1664	37.33	- 18		60
	.1664	35.43	- 50		
	.1727	39.43	-149		
	.1727	39.83	-184		75
	.1727	31.53	-216		
	.1674	30.43	-237		90
	.1743	15.03	-320		
	.1743	8.23	-320		105
	.1743	13.73	-320		120
	.1743	16.03	-320		135
	.1593	16.53	-320	(Equil.)	150
	.1721	3.13	+ 75	(Equil.)	195
	.2164	35.43	+ 75		
	.2449	51.93	+ 75		
	.2812	68.03	+ 75		
	.3251	76.53	+ 75		
	.3245	54.43	+ 75		
	.3135	31.63	+ 75		
	.2994	13.63	+ 75		
	.3053	0	+ 75		

LN$_2$ SEAL TEST DATA

MATERIAL	TEMPERATURE (°F)	INLET PRESSURE (PSI)	VALVE DOME LOAD FOR ZERO LEAKAGE (LBS)	STRESS (PSI)
TEFLON TFE				
	+ 70	200	8	460
	+ 70	400	22	1260
	-320	200	50	2590
	-320	400	65	3750
		Cycle Valve 100 Times		
	-320	200	50	2880
	-320	400	62	3560
	+ 70	200	8	460
	+ 70	400	21	1210
AF-E-124D				
	+ 70	200	12	700
	+ 70	400	12	700
	-320	200	40	2300
	-320	400	44	2500
		Cycle Valve 100 Times		
	-320	200	44	2500
	-320	400	40	2300
	+ 70	200	10	575
	+ 70	400	10	575

SOLVENT EXPOSURE TEST DATA

Material & Exposure Period AF-E-124D	Δ Wt Init. X 10^{-4} gr	Δ Wt 1 Week Dry X 10^{-4} gr	Δ Length Init. X 10^{-3} in	Δ Length 1 Week Dry X 10^{-3} in	Δ Thickness Init. X 10^{-3} in	Δ Thickness 1 Week Dry X 10^{-3} in
Distilled Water						
1 Hr	+ 5	0	- 10	- 8	+ 23	- 6
4 Hrs	+ 9	+ 2	+ 9	+ 5	+ 50	+ 8
24 Hrs	+ 20	+ 1	- 5	+ 5	- 5	+ 1
3 Days	+ 36	+ 11	- 13	+15	- 14	+ 8
7 Days	--	+ 13	+ 8	+18	+ 9	+ 5
14 Days	+ 82	+ 28	0	+17	- 4	-11
Freon TF						
1 Hr	+ 730	+ 287	+ 8	+13	+ 38	+17
4 Hrs	+1659	+ 677	+ 43	+20	+ 35	+30
24 Hrs	+6355	+1629	+320	+50	+171	+30
3 Days	+5719	+1640	+185	+46	+112	-26
7 Days	+5667	+ 934	+198	+32	+137	+30
14 Days	+5895	+1707	+145	+45	+101	+40
IPA						
1 Hr	+ 4	- 1	+ 21	+25	+ 3	+19
4 Hrs	+ 6	+ 16	+ 23	+24	+ 5	+16
24 Hrs	+ 4	+ 4	+ 16	+26	+ 4	+11
3 Days	+ 3	+ 5	+ 19	+50	+ 3	+40
7 Days	--	+ 6	+ 40	+45	+ 1	+ 9
14 Days	- 8	- 1	+ 5	+25	- 1	+10
Trichlor-Ethylene						
1 Hr	+ 12	+ 10	+ 5	+38	+ 1	+ 6
4 Hrs	+ 22	- 19	- 16	+20	+ 8	+ 5
24 Hrs	+ 55	+ 18	0	+30	- 7	+ 9
3 Days	+ 94	+ 40	+ 10	+20	+ 1	-14
7 Days	+ 544	+ 64	+ 16	+20	+ 1	+13
14 Days	+ 193	+ 109	0	+20	+ 2	+13

SOLVENT EXPOSURE TEST DATA

Material & Exposure Period Teflon TFE	Δ Wt		Δ Length		Δ Thickness	
	Init.	1 Week	Init.	1 Week	Init.	1 Week
	X 10^{-4}gr	X 10^{-4}gr	X10^{-3}in	X 10^{-3}in	X 10^{-3}in	X 10^{-3}in
Distilled Water						
1 hr	+ 4	+ 5	- 5	-22	+15	+ 9
4 Hrs	+ 3	- 2	- 5	- 5	+10	+ 5
24 Hrs	+ 2	- 5	-22	-20	+ 5	- 5
3 Days	+ 7	0	-11	-14	+ 5	0
7 Days	+ 11	- 9	+25	- 9	+ 1	+ 2
14 Days	- 10	- 5	--	- 8	+ 7	+ 4
Freon TF						
1 Hr	+ 14	0	+ 7	-15	-10	+ 3
4 Hrs	+ 40	+ 13	0	0	+ 9	+ 9
24 Hrs	+ 86	+ 37	-17	-13	+17	+ 1
3 Days	+150	+ 85	- 9	-13	+13	+ 2
7 Days	---	+149	+31	- 1	+17	+11
14 Days	+321	+219	+13	0	- 5	+ 4
IPA						
1 Hr	+ 1	- 6	+12	+ 2	+ 9	+19
4 Hrs	+ 3	- 1	+35	- 4	+33	+ 7
24 Hrs	+ 5	- 3	+32	- 2	+32	+13
3 Days	+ 6	- 1	0	- 3	+28	+21
7 Days	+ 5	- 64	+17	-12	- 6	0
14 Days	+ 11	- 4	- 9	- 1	+ 4	+ 2
Trichlorethylene						
1 Hr	+ 2	+ 2	+ 8	- 1	+ 3	+ 1
4 Hrs	+ 10	+ 7	+ 2	0	+13	+15
24 Hrs	+ 21	0	- 2	+ 2	+ 8	+ 3
3 Days	+ 32	+ 8	--	-15	--	+13
7 Days	+ 58	+ 50	+ 4	- 2	+16	+17
14 Days	+ 50	+ 15	+13	- 5	+ 5	+ 2

MECHANICAL PROPERTIES DATA FROM FLUID EXPOSURE TEST

AF-E-124D	EXPOSURE PERIOD																	
	1 HR			4 HRS			1 DAY			3 DAYS			7 DAYS			14 DAYS		
FLUID	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C
DISTILLED WATER	680	230	2620	630	200	2240	700	190	1960	650	210	2430	—	—	—	690	220	2470
FREON TF	590	210	2010	370	200	1420	—	—	—	—	—	—	320	230	1250	280	200	850
IPA	620	200	2160	700	200	2100	700	210	2490	680	210	2360	670	210	2440	610	220	2390
TRICH.	610	220	2580	—	—	—	690	230	2790	—	—	—	620	190	1650	540	220	1930
TEFLON TFE																		
DISTILLED WATER	1890	240	2430	2130	190	2420	2290	180	2420	2190	190	2450	2230	210	2580	2190	180	2410
FREON TF	2150	200	2480	2210	150	2340	2180	200	2530	1990	220	2430	2000	230	2450	2080	190	2330
IPA	1620	280	2150	2140	180	2350	2070	210	2350	1980	180	2180	2240	140	2280	2180	150	2310
TRICH.	2190	160	2320	2190	180	2400	2180	140	2260	2140	150	2220	2120	110	2160	2180	190	2360

CONTROL*		
A	B	C
665	190	1935

CONTROL*		
A	B	C
1820	190	2185

A - LOAD AT 100% ELONGATION (POUNDS)
B - ELONGATION AT RUPTURE (%)
C - ULTIMATE STRESS (PSI)

* AVERAGE OF 4 SPECIMENS

COMPRESSION SET TEST DATA

(Specimen Diameter 0.5 in. dia. X .25 In. Thick)

MATERIAL	TIME COMPRESSED	TEMP	INIT. THICKNESS	COMP. THICKNESS	FINAL THICKNESS AFTER RELEASE			
	Hrs.	°F	In.	In.	1/2 Hr	1 Hr	2 Hrs	80 Hrs
AF-E-124D								
	24	77	.267	.189	.252	.256	.256	.265
	100	77	.253	.194	.2315	.232	.232	.246
	24	160	.241	.187	.204	.205	.205	.210
	24	200	.256	.188	.207	.207	.207	.214
	100	200	.256	.188	.199	.202	.203	.210
TEFLON TFE								
	24	77	.258	.231	.236	.237	.237	.240
	175	77	.254	.228	.232	.232	.232	.232
	24	200	.258	.233	.234	.234	.234	.234
	100	200	.255	.219	.219	.219	.219	.220